

DEPARTMENT OF COMMERCE

BUREAU OF STANDARDS

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UNITED STATES GOVERNMENT MASTER SPECIFICATION FOR
SOAP, CAKE, GRIT

FEDERAL SPECIFICATIONS BOARD SPECIFICATION No. 33a

[Revised October 28, 1926]

This specification was officially promulgated by the Federal Specifications Board on July 3, 1922, for the use of the departments and independent establishments of the Government in the purchase of grit cake soap.

[The latest date on which the technical requirements of this revision shall become mandatory for all departments and independent establishments of the Government is January 28, 1927. They may be put into effect, however, at any earlier date after promulgation]

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I. GENERAL SPECIFICATIONS

There are no general specifications applicable to this specification.

II. TYPES

Grit cake soap shall be furnished in two types, as follows: Type A, for fine work, such as glass and enamel; type B, for scouring and scrubbing.

III. MATERIAL AND WORKMANSHIP

Grit cake soap shall be satisfactory for the purpose intended.

IV. GENERAL REQUIREMENTS

See Detail requirements.

V. DETAIL REQUIREMENTS

Failure to meet any of the following requirements will be cause for rejection.

1. TYPE A

Matter volatile at 105 to 110° C. shall not exceed 4 per cent. Deliveries which yield more than 4 per cent of volatile matter shall be rejected without further test.

Alkali as alkaline salts (total alkalinity of matter insoluble in alcohol), calculated as sodium carbonate (Na_2CO_3), shall not exceed 1 per cent.

Free alkali, calculated as sodium hydroxide (NaOH), shall not exceed 0.1 per cent.

Insoluble siliceous material shall be not less than 88 per cent nor more than 93 per cent.

The insoluble siliceous material shall consist of not less than 90 per cent of ground feldspar. All of the insoluble siliceous material shall pass through a No. 100 sieve, and the residue retained on a No. 200 sieve shall not exceed 5 per cent.

Rosin, sugar, and foreign matter shall not be present.

Anhydrous soda soap shall be within 1 per cent of the difference between 100 and the sum of the matter volatile at 105 to 110° C., insoluble siliceous material, and alkali as alkaline salts.

The cakes shall be well compressed and of a satisfactory degree of friability, which shall not be materially affected or lessened after immersion in or contact with water.

The material shall not scratch glass or enameled surfaces.

The material shall be unscented and shall be of a light gray or white color.

2. TYPE B

Matter volatile at 105 to 110° C. shall not exceed 5 per cent. Deliveries which yield more than 5 per cent of volatile matter shall be rejected without further test.

Alkali as alkaline salts (total alkalinity of matter insoluble in alcohol), calculated as sodium carbonate (Na_2CO_3), shall not exceed 3 per cent.

Free alkali, calculated as sodium hydroxide (NaOH), shall not exceed 0.1 per cent.

Insoluble siliceous material shall not be less than 75 nor more than 85 per cent.

The insoluble siliceous material shall be mainly quartz, and it all must pass through a No. 100 sieve.

Rosin, sugar, and foreign matter shall not be present.

Anhydrous soda soap shall be within 1 per cent of the difference between 100 and the sum of the matter volatile at 105 to 110° C., insoluble siliceous material, and alkali as alkaline salts.

The cakes shall be well compressed and of a satisfactory degree of friability, which shall not be materially affected or lessened after immersion in or contact with water.

The material shall be unscented and shall be of a light gray or white color.

VI. METHODS OF INSPECTION, TESTS, AND BASIS OF PURCHASE

1. SAMPLING

(a) No samples shall be submitted with bids.

(b) One cake shall be taken at random from not less than 1 per cent of the vendors' shipping containers, provided such containers contain not less than 50 pounds each. In the case of smaller containers, a cake shall be taken at random from each lot of containers totaling not to exceed 5,000 pounds. The total sample shall in all cases consist of not less than three cakes taken at random from separate containers. With very large lots, where the sample drawn as above will amount to more than 20 pounds, the percentage of packages sampled shall be reduced so that the amount drawn shall not exceed 20 pounds.

The inspector shall note whether the material meets the specification regarding compression and friability.

Wrap the individual cakes tightly in paraffined paper at once and seal by rubbing the edges with a heated iron. The inspector should accurately weigh each wrapped cake, record its weight and the date of weighing on the wrapper, place the wrapped cakes in an air-tight container, which should be nearly filled, seal, mark, and send to the laboratory for test. Samples should be kept cool until tested. The seller shall have the option of being represented at the time of sampling, and, when he so requests, shall be furnished with a duplicate sample.

2. METHODS OF TESTING

(a) **PRELIMINARY TESTS.**—Note the net weight, color, and odor of the cakes; also note whether the cakes are well compressed and the

friability before and after immersion in or contact with water. With type A, only, determine whether the material scratches glass or enameled surfaces, rubbing the material onto a moistened cloth and then rubbing the surface with the cloth, keeping the soap in contact with the glass or enamel.

(b) PREPARATION OF SAMPLE.—Break a cake of average weight and shave from the freshly broken surfaces sufficient soap for analysis. Mix and weigh out all portions for analysis promptly. Preserve the remainder in an air-tight container in a cool place.

When a determination shows nonconformity with specification, a duplicate shall be run.

(c) MATTER VOLATILE AT 105° C.—Weigh 5 g of the sample in a porcelain or glass dish, about 6 to 7 cm in diameter and 4 cm deep, dry to constant weight in an inert atmosphere at a temperature not exceeding 105 to 110° C. Report loss in weight as matter volatile at 105° C.

(d) FREE ALKALI OR FREE ACID.—Digest hot a 5 g sample with 100 cc of freshly boiled neutral ethyl alcohol (94 per cent or higher). Filter through a counterpoised filter paper neutral to phenolphthalein or a weighed Gooch crucible with suction into a dry weighed beaker, protecting the solution during the operation from carbon dioxide and other acid fumes. Wash the residue on the paper or in the crucible with hot neutral alcohol until free from soap. Titrate the filtrate, using phenolphthalein as indicator, with standard acid or alkali solution, and calculate the alkalinity to sodium hydroxide or acidity to oleic acid.

(e) MATTER INSOLUBLE IN WATER.—After filtering and thoroughly washing the residue from (d), extract and wash it thoroughly with hot water. Dry the filter and residue at 105 to 110° C. for three hours, cool, and weigh matter insoluble in water. The nature of this may be determined by further examination. The insoluble matter should be siliceous. The approximate amount of feldspar contained in the abrasive material of scouring soap (when such material is known to contain nothing but feldspar or quartz or a mixture of the two) may be determined by decomposing about 0.5 g of the abrasive material with hydrofluoric acid, taking up the residue in water and hydrochloric acid and determining the Al_2O_3 . This weight multiplied by 5.48 and divided by the weight of sample gives the approximate percentage of feldspar in the abrasive material. Feldspar may be identified and the relative amounts of feldspar and quartz roughly determined by means of the petrographic microscope.

(f) ALKALI AS ALKALINE SALTS (TOTAL ALKALINITY OF MATTER INSOLUBLE IN ALCOHOL).—Titrate the filtrate from the determination of matter insoluble in water with standard acid, using methyl orange as indicator. Calculate alkalinity to sodium carbonate (Na_2CO_3).

(g) **SIEVE TEST.**—Dry in an oven at 105 to 110° C. a No. 100 sieve and a No. 200 sieve, cool, and weigh accurately. Weigh an amount of soap containing 10 g of insoluble siliceous material (see VI, 2, (e)), transfer to a beaker, add about 200 cc of water, and digest on a steam bath about one hour to dissolve the soap and other soluble matter. Pour the solution through the No. 100 sieve, wash the insoluble material from the beaker onto the sieve with water, and wash with water until all but the particles too coarse to pass the No. 100 sieve have been washed through, catching all of the liquid and solid matter passing through the sieve in a clean beaker or dish. Dry the sieve for one hour at 105 to 110° C., cool, and weigh. Calculate the percentage of residue retained on the No. 100 sieve, based on the insoluble siliceous material. (If the material forms lumps or aggregates on washing with water, a camel's-hair brush may be used on the sieve.)

In a similar manner transfer the material that has passed through the No. 100 sieve to the No. 200 sieve and wash with water until all but the particles too coarse to pass the No. 200 sieve have been washed through. Dry the sieve for one hour at 105 to 110° C., cool, and weigh. Add the weight of the residue retained on the No. 100 sieve to the weight of the residue found on the No. 200 sieve and calculate the sum to percentage of residue retained on the No. 200 sieve, based on the insoluble siliceous material.

(h) **TOTAL ANHYDROUS SOAP.**—Evaporate the alcoholic solution obtained after filtering off and washing the matter insoluble in alcohol (VI, 2 (d)) to dryness, dry at 105 to 110° C. to constant weight. Report the result as total anhydrous soap.

(i) **ROSIN.**—A qualitative test for rosin may be made as follows: After decomposing a solution of the soap and separating the fatty acids, heat a small quantity of the latter with acetic anhydride, cool, place a few drops on a spot plate, and add a drop of H_2SO_4 (specific gravity=1.53) to this. A fugitive violet color indicates the presence of rosin.

(j) **SUGAR.**—A qualitative test for sugar may be made as follows: Add a decided excess of hydrochloric acid to a solution of the soap, heat on a steam bath for 15 minutes, cool, filter from fatty acids, and test a portion of the filtrate which has been neutralized with sodium hydroxide solution by boiling for two minutes with an equal volume of boiling Fehling solution. The formation of red cuprous oxide indicates the presence of sugar.

3. REAGENTS

(a) **STANDARD SODIUM HYDROXIDE SOLUTION.**—0.25 *N*, or about 10 g, sodium hydroxide dissolved in water and diluted to 1 liter. Standardize against Bureau of Standards benzoic acid.

(b) STANDARD SULPHURIC ACID SOLUTION.—0.5 *N*, or about 25.8 g, strong sulphuric acid (specific gravity=1.84) diluted to 1 liter. Standardize against standard sodium hydroxide solution VI, 3 (a).

(c) SULPHURIC ACID (SPECIFIC GRAVITY=1.53).—Mix 62.5 cc of strong sulphuric acid (specific gravity=1.84) with 61.5 cc of water.

(d) FEHLING SOLUTION.—(1) *Copper Sulphate Solution*.—Dissolve 34.639 g of copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in water and dilute to 500 cc.

(2) *Alkaline Tartrate Solution*.—Dissolve 173 g of Rochelle salts ($\text{NaKC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$) and 50 g of sodium hydroxide in water and dilute to 500 cc. Mix equal volumes of (1) and (2) immediately before use.

(e) METHYL ORANGE INDICATOR.—Dissolve 1 g of methyl orange in 1 liter of distilled water

(f) PHENOLPHTHALEIN INDICATOR.—Dissolve 1 g of pure phenolphthalein in 100 cc of 85 to 95 per cent ethyl alcohol.

4. BASIS OF PURCHASE

Material under each type shall be purchased by net weight.

VII. PACKING AND MARKING

Packing and marking shall be in accordance with commercial practice, unless otherwise specified.

VIII. NOTES

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